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Indian Standard

LIQUID SEDIMENTATION METHODS FOR DETERMINATION OF PARTICLE SIZE OF POWDERS

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NEW DELHI 110002

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LIQUID SEDIMENTATION METHODS FOR DETERMINATION OF PARTICLE SIZE OF POWDERS

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Indian Standard

LIQUID SEDIMENTATION METHODS FOR DETERMINATION OF PARTICLE SIZE OF POWDERS

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 13 August 1969, after the draft finalized by the Sieves, Sieving and Other Sizing Methods Sectional Committee had been approved by the Civil Engineering Division Council.

0.2 This standard describes liquid sedimentation methods for determining the particle size distribution in those fraction of powders which pass through a 75-micron IS Sieve.

0.3 Further, methods adopted for determination of size distribution should be such that conditions of tests are similar to conditions of use. For example, powders used in wet processes should be tested by liquid sedimentation. A dry powder used in air dispersed condition may be analysed by air elutriation. Such co-ordination, however, is not always necessary or possible.

0.4 The choice of the most suitable method for determination of particle size depends in addition to the factors stated under **0.3**, on factors like:

a) the purpose for which the analysis is required,
b) the size range and density of particles,
c) the important properties of powder,
d) the amount available for test, and
e) the method by which the gross sample has been collected.

0.5 In addition to the various methods described in the standard, there is another method called liquid column method with sedimentation accumulation. This method being not very popular in the country has not been covered in this standard.

0.6 In the formulation of this standard due weightage has been given to international co-ordination among the standards and practices prevailing in different countries in addition to relating it to the practices in the field in this country. This has been met by deriving assistance from the following publications:

B.S. 3406 : Part 2 : 1963 Methods for determination of particle size of powders. Part 2 Liquid sedimentation method. British Standards Institution.

JOGLEKAR (G D) and MARATHE (B R). Liquid for particle size analysis by sedimentation method. JSIR. 17A, No. 5; 1958, P 197-203.

1. SCOPE

1.1 This standard covers three methods by liquid sedimentation for quantitative determination of particle size distribution in sub-sieve range. The first two methods are used for the particle size down to about 3μ while the third method is applicable to the determination of particle size distribution in the range 3 to 0·01 micron.

2. TERMINOLOGY

2.0 For the purpose of this standard, the terms and definitions given in IS : 4124-1967* and the following shall apply.

2.1 Laboratory Sample — The portion of the gross sample which is delivered to the laboratory for the determination of particle size distribution.

2.2 Analysis Sample — The portion of the laboratory sample which is used in the size analysis apparatus.

3. BASIC PRINCIPLES

3.1 Sizing by methods of sedimentation are based on the rate of settling of well dispersed powder particles in a fluid. The rate of settling may be increased by increasing the gravity effect in a centrifuge. The finer particles which take long time to settle under the effect of gravity alone will settle quicker under centrifugal sedimentation.

3.2 Liquid sedimentation methods may be classified as incremental and cumulative. In the incremental method the concentration of powder particles is measured at a predetermined depth from the free surface of the suspension at a plane section across the sedimentation vessel. The weight of the particles extracted from the sedimentation column by incremental methods (Method given in **6** and **7**) determines directly the proportion by weight of the sample under test that consists of particles having a diameter less than that corresponding to the velocity of fall at the time of sampling.

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*Glossary of terms relating to powders.

In case of cumulative method the mean concentration of unsettled particles is measured over the whole distance from the surface level of the suspension to a known depth below the surface or alternately the total sediment of a known depth is measured.

3.3 Particle size is calculated using Stokes's equation in the following form:

$$d = \left[\frac{18\,\eta\, h}{(\sigma - \rho)\, g t} \right]^{\frac{1}{2}} \times 10^4$$

where

d = Stokes diameter of the particle in microns;

η = absolute viscosity of medium in poise;

h = distance in cm through which particle falls in time t in s;

σ = true density of particle in g/cm^3;

ρ = density of medium in g/cm^3; and

g = acceleration due to gravity in cm/s^2.

3.3.1 The magnitude of Reynolds number determines the upper limit of particle size. This number should not exceed 0·2 for limiting the error within 5 percent. The Reynolds number is given by:

$$(v\, d\, \rho/\eta) \times 10^{-4}$$

where

$v = h/t$, the free falling velocity.

The lower limit of particle size is the size that is not disturbed by convection caused by small changes in temperature within the fluid and by diffusion. The upper and lower limits of materials of different densities that may be measured by this method are given, for ready reference, in Appendix A.

4. SUSPENSION AND DISPERSION

4.1 The important consideration in the measurement of particle size is the selection of suitable liquid suspending medium. The important characteristics are that Reynolds number should not exceed 0·2. Further the liquid should be able to disperse the particles efficiently. Some suspending media require a small quantity of dispersing agent to avoid flocculation. The selection of dispersing agent is made by trial and error method. The available data on liquids suitable as suspension liquids along with suitable dispersing agents are given in Appendix B. Suspension should be thoroughly stirred after adding the dispersing agent. The degree of dispersion achieved may be assessed by the methods described in Appendix C.

5. PREPARATION FOR A SEDIMENTATION ANALYSIS

5.1 Dry the sample. Take a representative test sample by the method of quartering or by use of a sampler. Sieve on a 75-micron IS Sieve by wet method. Record the percentage of weight remaining on the sieve.

5.2 Select a suspending liquid which will not change the physical and chemical characteristics of the powder and will conform to the requirements of **4** (*see* Appendix B). Special care shall be taken to avoid convection which normally occurs if the density difference between powder particles and the suspending liquid is less than 0·5 g/cc.

NOTE 1 — Viscosity and density of the suspending liquid may be determined in accordance with P : 25 and P : 16 respectively of IS : 1448 - 1960*.

NOTE 2 — Density of the particles may be determined in accordance with IS : 2720 (Part III) - 1966†.

5.3 Add the suspending liquid slowly to the dry sample. If the suspending liquid does not readily wet the powder a drop or two of dispersing agent should be added to the dry test portion. Work the powder to a paste and then to a liquid suspension with some suitable implement taking care to avoid grinding of the sample. Make the volume to about 150 ml with the suspension liquid, subsequently diluting to the requirements of the method. Stir by a mechanical stirrer or by compressed air, if considered necessary. In the latter case remove the air from the suspension by subjecting it to reduced pressure.

5.4 Apparatus should be located in a draught and vibration free position in the laboratory and all liquids used as suspension media stored in the immediate vicinity so as to obtain ambient temperature.

5.5 The apparatus, sample and suspending medium shall be at the same temperature and the temperature shall not vary during the test by more than 1 deg C.

6. METHOD I—FIXED POSITION PIPETTE INCREMENTAL METHOD—PIPETTER AT THE TOP

6.1 Principle — The pipetting method involves withdrawal of suspension by means of a calibrated pipette after known intervals of time, the tip of pipette being at a fixed position in the sedimentation vessel. The depth h below the free surface of the suspension is noted after each withdrawal. After time t the sample contains only those particles with Stokes 'diameter' less than that of particles settling at rate h/t since all particles larger than this will have settled below the sampling point. Weights of the residue in the withdrawn suspension directly gives the cumulative weights of the particles smaller than those having free fall velocities greater than h/t. For obtaining reasonable weights of the withdrawn sample, the concentration of the suspension should be about 1 percent by volume. The concentration may be reduced to minimize the effects of flocculation if a stable dispersed suspension cannot be achieved. In this case it should be borne in mind that the results may be of dubious accuracy.

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*Methods of test for petroleum and its products.

†Methods of test for soils: Part III Determination of specific gravity.

6.2 Apparatus — Details of the apparatus are given in Fig. 1

6.2.1 *Sedimentation Vessel* — The glass sedimentation vessel should be of about 5 cm internal diameter having a graduated scale from 0 to 20 cm marked on the side of the vessel. The zero graduation mark should be at least 2·5 cm above the inside base of the vessel.

6.2.2 *Pipette* — A pipette is fitted with a two-way tap and side discharge tube. The capacity of the pipette to the graduation mark is normally 10 ml. A bell shaped dome is fused to the pipette with a ground glass joint to fit the neck of the sedimentation vessel. A small vent hole is made in this dome. The inlet to the pipette stem should be level with the zero mark on the sedimentation vessel. The stem from the pipette bulb to the sampling inlet is constructed of capillary glass tube with a nominal bore of 1 mm. The tube above the bulb should be about 4 mm bore.

6.2.3 *Ancillary Apparatus* — The following are the ancillary apparatus:

a) Transparent sided constant temperature bath of at least 15 litres capacity, into which the sedimentation vessel can be immersed to the graduation mark.

b) *Balance* — having sensitivity of not less than 0·1 mg.

c) *Drying oven* — capable of being maintained within a temperature range suitable for evaporation of suspending liquid and which does not affect the composition of the material.

d) *Wide mouthed weighing bottle* — of capacity not less than 20 ml.

6.3 Preparation

6.3.1 *Calibrations of Pipette* — Clean and dry the apparatus. Then fill the sedimentation vessel with water up to about 20 cm level. Set the tap of the pipette to sampling position and suck water into the bulb to the level of the graduation mark by means of a rubber tube. Allow the water to drain into a weighed weighing bottle by turning the tap to discharge position. Apply pressure through the rubber tube to blow any water remaining in the bulb and the discharge tube into the bottle. Calculate the volume V_p of the pipette from the weight of the water in the weighing bottle.

6.3.2 *Temperature* — Maintain the requirements as given in **5.5**.

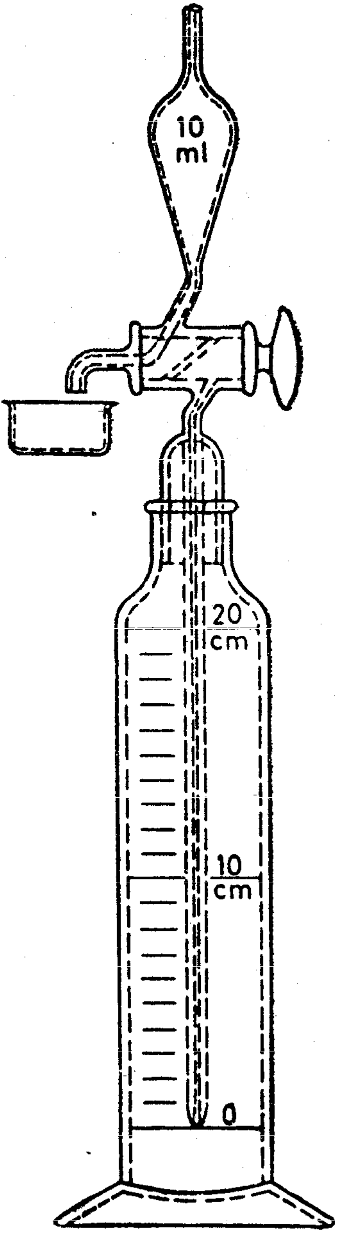


FIG. 1 FIXED POSITION PIPETTE INCREMENTAL METHOD — PIPETTE AT THE TOP

6.3.3 *Analysis Sample* — Make analysis sample as in **5**. A concentration of not more than 1 percent by volume should be used.

6.3.4 *Withdrawal Time Calculation* — Calculate the withdrawal time t_1 for the first fraction using Stokes's Law in the following form:

$$t_1 = \frac{18\,\eta\,h}{(\sigma - \rho)\,g\,d^2_1} \times 10^8 \text{ seconds}$$

where

η = absolu e viscosity of medium in poise;

h = mean depth at which extractions are made in cm, which may conveniently be 10 or 20 cm ± 1 mm;

σ = true density of particle in g/cm^2;

ρ = density of medium in g/cm^3;

g = acceleration due to gravity in cm/s^2; and

d_1 = limiting Stokes diameter for the first fraction, which is preferably 53 microns and shall not exceed 75 microns.

6.4 Sedimentation

6.4.1 *Procedure*

6.4.1.1 Transfer the prepared suspension without any loss into the sedimentation tube. Then fill up to the graduation mark with the suspending liquid. Place the sedimentation vessel into the constant temperature bath or water bath at room temperature till it attains the temperature of bath. Record the temperature. Then mix the contents of the sedimentation tube by inverting the vessel many times. Care should be taken to close the vent hole in the dome when inverting the sedimentation vessel. Immediately after mixing start the stop watch.

6.4.1.2 Withdraw from the suspension by means of the pipette a series of 10 ml fractions at depth h (for example, 10 or 20 cm ± 1 mm) at increasing time intervals starting with t_1 as in **6.3.4** and thereafter the 2 : 1 progression so that limiting diameters are in the ratio $\sqrt{2:1}$ progression. If the intervals of time are not precisely maintained use of equation given under **6.5.1** shall be made.

6.4.1.3 The time to fill the pipette should be about 20 s. Set the tap of the pipette to sampling position. Apply suction to withdraw a fraction from the sedimentation tube 10 s before the appropriate time, care being taken to avoid too rapid entry of liquid into the pipette, with consequent eddying of the suspension. The tap is then closed and pipette withdrawn. The suspension is discharged into a tared weighing bottle. When the pipette bulb has drained, remove the rubber suction tube run 5 to 7 ml of pure suspending liquid from another 10 ml pipette into the bulb of the sedimentation pipette to wash into the weighing bottle any particles adhering to the surface of the pipette.

6.4.1.4 Since depth of immersion of pipette decreases after each fraction is withdrawn, mean depth should be used in calculations. Suppose that initial depth of immersion of pipette is h_i and after that withdrawal of sample is h_f. Then the mean depth of immersion of pipette is $(h_i + h_f)/2$.

6.4.1.5 *Assay of fraction* — Evaporate each fraction in its weighing bottle to dryness in an oven maintained at a temperature suitable to the particular suspending liquid and the material under test [*see* **6.2.3** (c)] and then cool in a desiccator. Determine the weight W_n of solid material of each fraction by weighing the bottle and its contents and then subtracting the weight of the bottle. Allowance for the weight of the dispersing agent may also be made, if necessary.

6.5 Calculation of Results

6.5.1 *Calculation of Limiting Stokes Diameter* — When the time of sampling is other than that specified in **6.4.1.2**, calculate the limiting Stokes diameter d_n corresponding to each of the time intervals t_n ($n = 1, 2, 3$, etc) from the following equation:

$$d_n = \left(\frac{18\, \eta\, h}{(\sigma - \rho)\, g t_n} \right)^{\frac{1}{2}} \times 10^4 \text{ microns}$$

6.5.2 *Calculation of Cumulative Percentage Undersize* — The cumulative percentage P_n smaller than the limiting diameter d_n for each time interval t_n from the weights W_n of the fractions is given by the following:

$$P_n = \frac{W_n \times V}{W_s \times V_p} \times 100$$

where

W_n = weight of fraction in g,
V = volume of sedimentation vessel to the mark ml,
W_s = weight of test portion in g, and
V_p = volume of pipette in ml.

6.6 Repeat Tests — Repeat the procedure using the same laboratory sample. The proportion by the weight less than the same limiting diameter should not differ by more than 2 percent.

6.7 Report — Plot the results of the analysis micron sizes as abscissae and the percentages undersize as ordinates. Draw a smooth curve and from the curve choose cumulative percentages corresponding to the series required.

6.7.1 The report shall state:

a) method used,

b) the suspending liquid,

c) the dispersing agent,

d) the volume concentration used, and

e) the density of the particles.

7. METHOD 1 — FIXED POSITION PIPETTE INCREMENTAL METHOD — PIPETTE AT SIDE

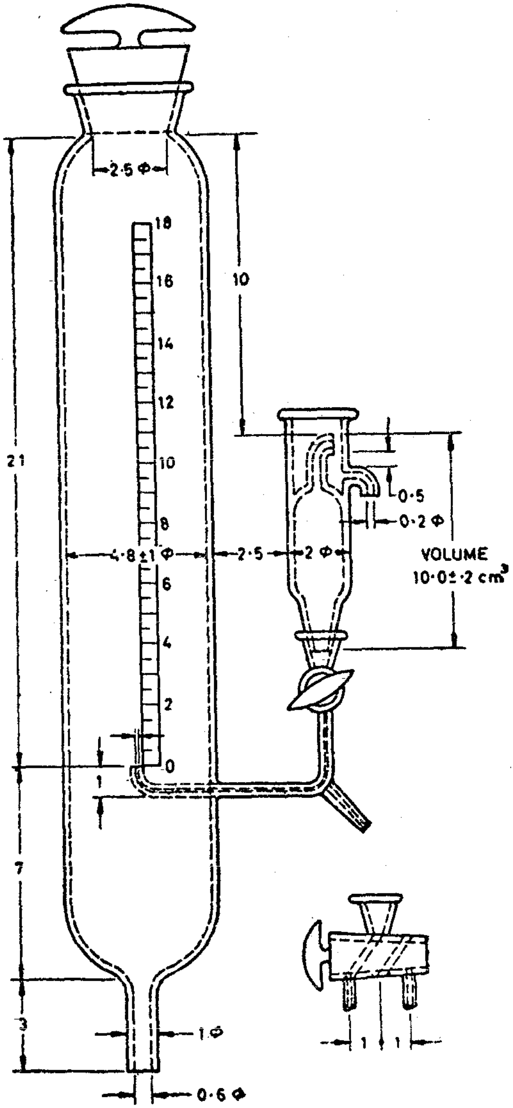
7.1 Principle — *See* **6.1**.

7.2 Apparatus — Details of the apparatus are given in Fig. 2.

7.2.1 *Sedimentation Vessel* — The internal diameter of the sedimentation vessel should be about 4·8 cm. The length of the vessel is about 28 cm having a graduated scale from 0 to 18 cm marked on the side of the vessel. This mark should be at least 5 cm above the bottom of the vessel as shown in Fig. 2. The zero mark is at the level at which the sample is withdrawn. At the bottom there is an outlet to drain the liquid or suspension. Compressed air can be passed through this outlet when necessary. A ground glass stopper with an air vent is provided to close the top of the vessel. The top of the vessel is about 3 cm above the 18 cm mark on the scale.

7.2.2 *Pipette* — A 10 ml pipette with a two way stop cock (pipette, preferably detachable from the stop cock) at the bottom and a side discharge tube is fixed on the side of the sedimentation vessel. The sample could be withdrawn from the sedimentation vessel by opening the cock connecting sedimentation vessel and the pipette. The sample is withdrawn up to a mark shown on the pipette to indicate that the volume of liquid in the pipette is 10 ml. As soon as the liquid is 10 ml in the pipette the cock is turned to drain the liquid into a weighed bottle. The bore of the tube that withdraws the sample to the pipette from the sedimentation vessel should be of nominal diameter of 2 mm. About 20 seconds should be allowed to fill the pipette.

7.3 Calibration of Pipette — The sedimentation tube including the pipette is cleaned and dried. The sedimentation vessel is then filled with distilled water. The stop cock on the pipette is opened in the sampling position and water is allowed to run into the pipette until it just overflows. The stop cock is reversed to the discharge position and water is collected in a tared weighing bottle. The correct volume of the pipette is determined from the weight of water divided by the density of the water at that temperature.



All dimensions in centimetres.

Fig. 2 Fixed Position Pipette Incremental Method—Pipette at Side

7.4 Temperature — *See* **5.5**.

7.5 Preparation of Analysis Sample — Procedure for preparation of analysis sample is same as given in **5**. A concentration of not more than one percent by volume should be used.

7.6 Withdrawal Time Calculation — *See* **6.3.4**.

7.7 Procedure — The sedimentation vessel is cleaned and then the outlet is closed by a pinch cock on a rubber tube connected to the outlet. The suspension as described in **7.5** is carefully poured into the sedimentation vessel. The beaker is cleaned many times with the sedimentation liquid and the liquid is poured into the sedimentation vessel to bring all the traces of powder from the beaker into the sedimentation vessel. Then pour more liquid to make the volume up to the graduation marked 18 cm. If necessary connect a pressure pump to the exit tube provided at the bottom of the sedimentation vessel. The pump is started and controlled rate of air is allowed to enter the sedimentation vessel by opening the pinch cock. Air bubbling causes very intense agitation of suspension. After about three minutes agitation, stop passing air and immediately start the stopwatch. A series of samples (fractions) are then withdrawn through the pipette. Rest of the porcedure is as given in **6.4**.

7.8 Assay of Fractions — *See* **6.4.1.5**.

7.9 Calculation of Results — *See* **6.5**.

7.10 Repeat Tests — *See* **6.6**.

7.11 Report — *See* **6.7**.

7.12 Example — A worked out example illustrating method described in **7** is given in Appendix D.

8. METHOD II — CENTRIFUGAL SEDIMENTATION METHOD

8.1 Principle — Measurement of particles smaller than 2 microns is difficult by sedimentation under gravity on account of very long time needed for sedimentation. In addition to this, convection and diffusion effect on particles smaller than 2 microns may be significant. These effects may be reduced by increasing the gravity effect by centrifugal acceleration. Under a centrifugal force, Stokes equation may be written as:

$$\frac{\pi}{6} d_e^3 (\sigma - \rho) \omega^2 R = 3 \pi \eta d_e (dR/dt)$$

where

ω = angular velocity. The other symbols have the same meaning as explained in **3.3**. Integrating between the limits R_1 and R_2 we get

$$d_e = [18\,\eta\,l_n\,(R_2/R_1)/(\sigma - \rho)\,w^2\,t]^{\frac{1}{2}}$$

$$\text{or } d_e = k/\,(t)^{\frac{1}{2}}$$

where

$$k = [18\,\eta\,l_n\,(R_2/R_1)/(\sigma - \rho)\,\omega^2]^{\frac{1}{2}}$$

Rate of accumulation of sedimented particles is measured. The suspension is centrifuged for a predetermined time with a known velocity. The samples are then separated into two components; the sediment which is normally packed firmly at the bottom of the tube; and the suspension that is those particles still suspended in the liquid medium. Dry the two components and weigh them accurately. A curve is drawn between the percentage of sedimented powder and particle size in microns. This is converted to size distribution curve by performing a tangent drawing operation.

8.2 Apparatus — The centrifuge is composed of the following:

a) Equipment to impart angular velocities up to 5 000 rev/min,
b) Accurate means to measure speed of rotation,
c) Good control on speed (at least for 30 min),
d) A braking device to stop the centrifuge in the shortest time, and
e) A tube of 5 cm length situated about 15 cm from axis.

8.2.1 The accessories should include a centrifuge holding eight chromium plated metal tubes or glass tubes of size 4·0 by 9·2 cm. Tubes are covered with metal caps to prevent evaporation and other disturbances during sedimentation. For the measurement of speed a universal hand tachometer or any other device may be used. Acceleration and retardation should take the shortest possible time. A voltage regulator may be used with the mains to maintain steady revolution.

8.3 Using the sample, find the particle size distribution by the method described in **7**. Note the size of the smallest particles determined by the method. A worked out example is given in Appendix E.

8.4 Preparation of Analysis Sample — Choose suitable sedimentation liquid (*see* Appendix B). Weigh the sample accurately to 0·000 1 g. Prepare 300 ml volume of the suspension in the sedimentation liquid as directed in **5**. The concentration of the powder in the suspension should not be more than 1 percent by volume.

8.5 Temperature — The experiment should be done in a room where temperature variations during the period of experiment are negligible (permissible variation 1°C).

8.6 Time Calculation — For a given particle size, the time of sedimentation may be computed from the following equation (symbols are the same as in **8.1**):

$$d_e = \frac{k}{(t)^{\frac{1}{2}}}$$

8.7 Procedure — The procedure is as given in **8.7.1** and **8.7.2**.

8.7.1 Transfer the whole of the suspension without loss to a flat bottomed cylinder of 30 cm length. Then it is kept in a water jacket after a final shaking so as to disperse the powder uniformly in the liquid. Calculate the time using equation in **6.3.4** for particles of smallest size determined by the method given in **7**. This size shall conform to **3.3.1** to settle to a depth of 18 cm from the surface of the liquid in the vessel. Allow the suspension to settle for the above calculated time. Decant the supernatant liquid by withdrawal by suction from a tube immersed to a fixed depth or using a 'broken' siphon immersed to the required depth. Without disturbing the liquid below the calculated level of 18 cm, transfer the remaining suspension and sediment into an evaporating vessel and determine the weight of sediment by drying and weighing. The concentration of the particles in the decanted liquid is then determined by dividing the difference in weight of original sample taken and the weight determined above by the volume of decanted liquid. Concentration of the decanted suspension may also be found by taking 10 ml of thoroughly mixed decanted liquid in a weighed crucible and then drying and weighing it.

8.7.2 Pour 5 ml of the thoroughly mixed decanted liquid into each of the centrifuge tubes. Take readings at different timings keeping the speed of the centrifuge constant. At each interval stop the centrifuge. Remove from it one tube, decant the liquid from it and then dry and weigh the tube. Use equation in **8.1** to find the particle size in the supernatant liquid taken out according to **8.7** (a) particles will be lost to the liquid column of length determined by equation given in **3.3**. The depth of column h_x not having a particular particle size d_x is given by the following relation:

$$h_x = \left[\frac{d_x}{d_o} \right]^2 h_o$$

where

d_o = smallest size determined by the method given in **7**, and
h_o = 18 cm.

Hence the actual weight of particles W_{xa} of size d_x that would have been in 18 cm column if they were not missing in h_x is given by

$$W_{xa} = W_x \, h_o / (h_o - h_x)$$

where

W_{xa} = observed weight in grams of particles of size d_x.

The powder in the tube contains particles of larger size than the size calculated for the speed and the time and also contains some smaller particles since some particles had to travel a smaller distance. In order to determine the actual particle size odens methods of tangential intercept is used. In this method percentage of sedimented material is plotted against $1/d_e^2$. Tangents to the curve at different values of $1/d_e^2$ are drawn and the intercepts on vertical axis represent the percent undersize of the powder particles for the corresponding diameter.

8.8 Repeat Tests — *See* **6.6**.

8.9 Report — *See* **6.7**.

8.10 Example — A worked out example illustrating Method II is given in Appendix E.

9. CORRELATION OF RESULTS FROM DIFFERENT METHODS OF SIZE DETERMINATION

9.1 A number of methods such as sieving, sedimentation, elutriation microscope, etc, are used for determination of particle size. No single method is applicable to the whole range of sizes 1 000-1 microns so that, if a powder being analysed is composed of particles of a wide range of sizes, it is necessary to combine the analysis by two or more different methods in order to establish the distribution in full. The correlation factors recommended to be adopted to convert particle diameters determined from one method to another are given in Appendix F.

APPENDIX A

(*Clause* 3.3.1)

UPPER AND LOWER LIMITS OF PARTICLE SIZES FOR DIFFERENT DENSITIES OF MATERIAL

A-1. The lower and upper limits of particle size of powders of a given density are given below. In this calculation viscosity of the fluid is taken as 0·01 poise and the density of fluid as 1·00 g/cc, Reynolds number is taken as 0·2 for the upper limit. For a material of density 2·00 g/cc the lower limit of particle size is taken as 3·0 microns and for other materials the particle size varies in the same ratio as in the case of upper limit.

Material	*Density*	*Diameter in microns*	
	g/cc	Upper limit	Lower limit
A	2	72	3·0
B	4	50	2·1
C	8	37	1·6

APPENDIX B

(*Clauses* 4.1, 5.2 *and* 8.4)

Sl No.	*Material*	*Liquid and Dispersing Agent**
1)	Alumina	Water; water (calgon, 0·1 percent); water (sodium tartrate, 0·1 percent); dilute hydrochloric acid (*p*H of suspension adjusted to 3); carbon tetrachloride
2)	Alumina dust	Water (sodium oxalate)
3)	Alundum	Water
4)	Aluminium powder (sp. gr. 2·5)	Isopropanol; chloroform
5)	Anthracite	Water (perminal BX, 0·1 percent)
6)	Barytes	Water (sodium pyrophosphate, 0·001-0·005 mol/litre)
7)	Barium carbonate (precipitated)	Methyl alcohol
8)	Barium strontium carbonate	Water-ethyl alcohol mixture
9)	Bronze powder	Cyclohexanol
10)	Chromic oxide	Water (sodium pyrophosphate, 0·001-0·005 mol/litre)
11)	Colours (mineral)	Water (potassium citrate, 0·1 mol/litre)
12)	Carbon black	Water (Aerosol OT, 1 percent); gallotannic acid
13)	Calcium carbonate	Water; xylene
14)	Calcium carbonate (precipitated)	Water (sodium pyrophosphate, 0·005 mol/litre)
15)	Calcium arsenate	Ethyl alcohol-water (1 : 1)
16)	Cryolite	Glycerol-water (1 : 4; v/v)
17)	Calomel	Cyclohexanol

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*Where a dispersing agent is used, it is given along with its concentration within parenthesis.

Sl No.	*Material*	*Liquid and Dispersing Agent**
18)	Coal	Water (Aerosol OT, ½ percent); water (sodium linoleate and sulphonated loral, 1 percent); ethyl alcohol (calcium chloride, 0·1 mol/litre); ethyl alcohol; ethyl industrial absolute alcohol; xylene; alcohol-water mixture (1 : 1) acetone or petroleum
19)	Clay	Water; water (sodium oxalate); distilled water (sodium silicate, 2 percent)
20)	Copper	Water; acetone; rape oil; rape oil + acetone; soyabean oil + acetone (1 : 1)
21)	Cobalt metal	Ethyl alcohol, 95 percent; rape oil + acetone
22)	Chalk	Water (sodium silicate, 2 percent); water (potassium citrate, 0·1 g/mol); acetone or petroleum
23)	Chalk (precipitated)	Isopropanol
24)	Cocoa	Isobutyl alcohol; diethyl ester of phthalic acid
25)	Coke	Water (perminal BX, 0·1 percent); distilled water (tannic acid, 0·5 g/litre and ammonia, 0·88-3·5 ml/litre)
26)	Cement	Water; kerosene (oleic acid) benzene; isopropanol; ethylene glycol (calcium chloride); absolute alcohol (anhydrous calcium chloride); methyl alcohol (sodium pyrophosphate); butyl alcohol
27)	Alumina cement	Glycol (cobalt chloride); ethylene glycol (cobalt chloride)
28)	Portland cement	Ethyl alcohol; butyl alcohol; benzyl alcohol; glycol; castor oil; paraffin oil

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*Where a dispersing agent is used, it is given along with its concentration within parenthesis.

Sl No.	*Material*	*Liquid and Dispersing Agent**
29)	Diamond powder	Olive oil; water (gelatine, 0·1-0·2 percent and sodium carbonate)
30)	Dicalcium phosphate	Water and alcohol
31)	Felspar	Water (sodium oxalate)
32)	Flint	Water (sodium oxalate)
33)	Flours	Isobutyl alcohol; isobutyl alcohol + diethyl phthalate; diethyl ester of phthalic acid; petrol
34)	Fluorite	Water (gelatine, 0·1-0·25 percent and sodium carbonate)
35)	Flourspar	Dilute nitric acid (0·002 N); methyl alcohol (potassium chloride, 0·001M)
36)	Fly ash	Water
37)	Glass powder or spheres	Water; water + ethylene glycol; butyl alcohol; methanol
38)	Graphite	Water (Aerosol OT, ½ percent); ammonia, 0·880-3·5 mil/litre and sodium linoleate; distilled water (tannic acid, 0·5 g/litre); ethyl alcohol
39)	Glass powder (colloidal)	Lignin
40)	Gypsum	Alcohol (calcium chloride, 0·1 mol/litre); glycol (cobalt citrate); ethylene glycol (cobalt citrate)
41)	Haematite	Water
42)	Hydrated lime	Ethyl alcohol; isopropanol
43)	Limenite	Water
44)	Iron and iron alloys	Rape oil + acetone
45)	Iron metal	Soyabean oil + acetone (1 : 1 by volume)

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*Where a dispersing agent is used, it is given along with its concentration within parenthesis.

Sl No.	*Material*	*Liquid and Dispersing Agent**
46)	Kaolin	Water (sodium pyrophosphate, 0·005 mol/litre and sodium silicate, 0·1 percent); water (ammonia, a few drops, if necessary)
47)	Lead	Acetone; water
48)	Lead oxide	Xylene; ethy alcohol; rectified spirit
49)	Lignite	Isobutyl alcohol; diethyl ester of phthalic acid
50)	Limestone dust	Water (sodium pyrophosphate, 0·005 mol/litre and sodium silicate, 0·2 percent); distilled water (sodium silicate, 2 percent and potassium citrate, 0·1 mol/litre); glycerol + water (1 : 4 by volume)
51)	Lithophone	Glycerine
52)	Manganese dioxide (pyrolusite)	Water (sodium pyrophosphate, 0·001-0·005 mol/litre)
53)	Magnesite (burnt)	Ethylene glycol
54)	Magnetite powder	Water; ethyl alcohol; methyl alcohol; nitrobenzene
55)	Mineral colours	Water (potassium citrate, 0·1 mol/litre)
56)	Moulding sand	Distilled water (sodium hydroxide)
57)	Molybdenum powder	Ethyl alcohol; acetone; glycerol; glycerol + water
58)	Nickel powder	Rape oil + acetone
59)	Phosphorus	Water (Daxad 23, 0·02 percent and potassium silicate, 0·1 percent)
60)	Pigments	Water (sodium pyrophosphate); isopropanol
61)	Plaster (Stucco)	Alcohol-glycol (potassium citrate); water (potassium citrate, 0·1 mol/litre)

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*Where a dispersing agent is used, it is given along with its concentration within parenthesis.

Sl No.	*Material*	*Liquid and Dispersing Agent**
62)	Precipitated magnesium carbonate	Acetone
63)	Pumicite	Water
64)	Pulp	Water (sodium silicate, 1 part in 4 000 parts of liquid)
65)	Quartz	Water (sodium oxalate)
66)	Red lead	Paint prepared in linseed oil and dispersed in white spirit (aluminium stearate)
67)	Red phosphor (4% concentration)	Methylated spirit
68)	Sand	Water; distilled water (sodium silicate, 2 percent); butyl phthalate + alcohol
69)	Silica	Water; water (sodium pyrophosphate, 0·133 percent, 0·005 mol/litre, sodium oxalate, N/100 and Calgon, 0·1 percent); water + alcohol (1 : 1); xylene
70)	Silicates	Water (sodium pyrophosphate, 0·005 mol/litre); water + ethyl alcohol (1 : 1)
71)	Sillimanite	Water + ethyl alcohol (1 : 1); water (sodium pyrophosphate, 0·005 mol/litre)
72)	Shales	Alcohol (calcium chloride)
73)	Slag (cement)	Isopropanol; water
74)	Starch	Isobutyl alcohol; diethyl ester of phthalic acid; isobutyl alcohol + diethyl phthalate
75)	Soils	Water; water (sodium oxalate N/100 and 2 percent); butyl phthalate + alcohol

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*Where a dispersing agent is used, it is given along with its concentration within parenthesis.

Sl No.	*Material*	*Liquid and Dispersing Agent**
76)	Sugar	Isobutyl alcohol; diethyl ester of phthalic acid
77)	Sulphides	Water (saponin)
78)	Tin	Butyl alcohol
79)	Titanium	Water (Calgon, 0·1 percent)
80)	Tricalcium phosphate	Water
81)	Tungsten	Glycerol; acetone + rape oil; ethyl alcohol
82)	Tungsten carbide	Oil
83)	Zinc	Ethyl alcohol; butyl alcohol; acetone
84)	Zinc oxide (pigment)	Water (sodium hexametaphosphate, 0·5-1·0 g/litre)
85)	Zircon	Water; water (sodium pyrophosphate, 0·005 mol/litre); water + alcohol (1 : 1)
86)	Zirconium metal	Methyl alcohol (hydrochloric acid, N/100)

APPENDIX C

(*Clause* 4.1)

TESTS FOR DISPERSION

C-1. CRITERIA

C-1.1 A practical test is to carry out sedimentation analysis with different suspending agents and to accept the results showing the greatest proportion of fines. A list of a few suggested media, suspending agents and wetting agents is given in Appendix B. Any combination which foams on stirring shall be rejected, since this may cause separation of the fine particles.

C-1.2 The following tests are used for testing dispersion of the particles in the fluid. In certain cases it may not be necessary to carry out all the tests mentioned below. However at least two tests should be carried out to ensure efficient dispersion:

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*Where a dispersing agent is used, it is given along with its concentration within parenthesis.

a) Rheological behaviour,
b) Microscopical examination,
c) Qualitative sedimentation, and
d) Quantitative sedimentation.

The first is a rapid test, which enables possible media and dispersing agents to be selected from those available. The suitability of the selected media and dispersing agents may be checked, if desired, by microscopic examination, but the final tests should be performed by sedimentation and observed both the qualitatively and quantitatively, especially in the case of unfamiliar materials.

C-2. RHEOLOGICAL BEHAVIOUR

C-2.1 A small quantity, 1 to 2 ml in bulk, of the powder is placed on a glass plate, and the medium containing the dispersing agent under test is added dropwise, the mixture being worked and kneaded with a flexible spatula or palette knife. Addition of the medium is continued until it is just possible to bring all the material together into a single coherent mass. If, on taking up this mixture on the spatula, it flows freely in long syrupy threads, and has marked dilatant* properties (for example starch in water) the dispersion is probably good. A less good but still generally satisfactory dispersion flows only on tapping the spatula with the finger nail when the blade is held horizontally. Flocculated mixtures, on the other hand, do not flow even on tapping but roll up on the spatula blade into a characteristic scroll-lime form (for example, heavy magnesium oxide in water) on the glass plate instead of spreading out into a glassy pool they appear dull and pasty, even after tapping.

C-3. MICROSCOPICAL EXAMINATION

C-3.1 A small drop of the suspension as prepared in the sedimentation apparatus, at the concentration to be used, is placed on a microscope slide, and a cover glass is gently lowered over it. Examination of all parts of the slide under an adequate magnification will show the particles to be entirely separate and free from each other in a good dispersion, while a poorly dispersed or flocculated sample will show characteristic chains and aggregates of particles.

C-3.1.1 Care should be taken with suspensions in mobile liquids, while lowering of the cover slip into the suspension so as to minimize preferential removal of the longer particles to the edge of the cover slip. Otherwise these particles which have smaller particles attached to them may give false idea of the degree of dispersion.

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*Dilatancy is the power of a suspension substantially to increase its viscosity on stirring and to resume its more fluid state on standing.

C-4. QUALITATIVE SEDIMENTATION (PRELIMINARY OBSERVATIONS)

C-4.1 Suspensions of the powder (approximately 2 percent by volume but in concentration not less than that to be used in the particle size analysis) are prepared in the selected media, and their behaviour is observed on standing. Good dispersions settle out less rapidly than suspensions which are wholly or partly flocculated and show no sharp line of demarcation between clear liquid and the turbid lower layers as settling proceeds. The appearance of the sediment itself is also a guide, as a good dispersion will yield a rigid sediment of minimum volume, while a flocculated suspension will produce a relatively large volume of sediment which will move like a liquid on tilting the tube. In a tilted tube a flocculated dispersion will also give a visible deposit on the wall.

C-5. QUANTITATIVE SEDIMENTATION (TESTS ON FINAL DISPERSION)

C-5.1 The quantitative test of good dispersion and freedom from mass convection effects is that the proportion of undersize should not be significantly increased by reducing the concentration of powder by a factor of 2. In no case should the initial concentration be greater than 2 percent by volume.

APPENDIX D

(*Clause* 7.12)

PARTICLE SIZE DISTRIBUTION OF ALUMINA POWDER

Liquid used: Water 450 ml
Dispersing agent: Sodium tartrate (0·1%)
Weight of powder: 0·9 g
Volume of pipette: 10 ml

Room temperature: 27°C
Density of alumina: 3·03 g/ml
Alumina powder in 10 ml : 0·020 0 g
Density of water at 27°C = 0·996 5 g/ml

CRUCIBLE No. (1)	WEIGHT OF CRUCIBLE (g) (2)	WEIGHT OF CRUCIBLE + SAMPLE (g) (3)	WEIGHT OF SAMPLE (g) (4)	TIME (*Min*) (5)	MEAN DEPTH OF IMMERSION OF PIPETTE h (cm) (6)	$\left(\frac{h}{t}\right)^{\frac{1}{2}}$ (7)	SIZE MICRON (8)	MEAN SIZE MICRON (9)	CUMULATIVE PERCENTAGE (10)	PERCENTAGE (11)
										2·0
1	12·765 4	12·695 0	0·019 6	½	17·73	5·955	68·89		98·0	
								58·44		3·5
2	12·723 2	12·742 1	0·018 9	1	17·18	4·145	47·98		94·5	
								40·68		6·0
3	12·624 5	12·042 2	0·017 7	2	16·63	2·883	33·38		88·5	
								28·30		9·5
4	12·547 6	12·563 4	0·015 8	4	16·08	2·004	23·21		79·0	
								19·67		24·0
5	12·821 2	12·832 2	0·011 0	8	15·53	1·393	16·13		55·0	
								13·66		7·5
6	12·729 2	12·737 7	0·009 5	16	14·98	0·968	11·20		47·5	
								9·49		6·0
7	12·452 1	12·460 4	0·008 3	32	14·43	0·672	7·78		41·5	
								6·58		9·5
8	12·521 7	12·528 1	0·006 4	64	13·88	0·466	5·39		32·0	
								4·79		4·0
9	12·872 9	12·878 5	0·005 6	128	13·33	0·362	4·19		28·0	
								3·39		12·0
10	12·712 3	12·715 5	0·003 2	256	12·78	0·224	2·59		16·0	
								1·30		16·0

APPENDIX E

(*Clauses* 8.3 *and* 8.10)

PARTICLE SIZE DISTRIBUTION OF CLAY (COARSER PART)

Liquid used: Water 450 ml
Dispersing agents: Sodium hexa meta phosphate (few drops)
Weight of clay taken = 4·5 g
Density of clay = 2·42 g/cm³

Volume of pipette = 10 ml
Temperature of room = 27°C
Density of water at 27°C = 0·996 5 g/cm³
Clay in 10 ml = 0·100 0 g

Crucible No.	Weight of Crucible (g)	Weight of Crucible + Sample (g)	Weight of Sample (g)	Time (*Min*)	Mean Depth of Immersion of Pipette cm	$(h/t)^{\frac{1}{2}}$	Size Micron	Mean Size Micron	Cumulative Percentage	Percentage
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)
1	12·765 4	12·863 4	0·100 0	0·5	17·73	5·955	82·39		100	0
								69·87		0
2	12·723 2	12·823 2	0·100 0	1	17·18	4·145	57·35		100	
								48·62		0
3	12·624 5	12·724 5	0·100 0	2	16·63	2·883	39·89		100	
								33·82		0
4	12·546 7	12·646 7	0·100 0	4	16·08	2·004	27·74		100	
								23·51		0
5	12·821 2	12·921 2	0·100 0	8	15·53	1·393	19·28		100	
								16·33		0
6	12·729 2	12·829 2	0·100 0	16	14·98	0·968	13·39		100	
								11·34		0
7	12·452 1	12·512 1	0·100 0	32	14·43	0·672	9·29		100	
								7·87		8
8	12·521 7	12·613 7	0·092 0	64	13·88	0·466	6·45		92	
								5·73		19
9	12·872 9	12·945 9	0·073 0	128	13·33	0·362	5·01		73	
								4·05		15
10	12·713 2	12·770 3	0·058 0	256	12·78	0·224	3·09*		58	
								1·54		58
										100

*Actual size 2·943.

NOTE — Cumulative percentage of particles below 2·943 μ is 58.

(*Continued*)

(*Appendix* E — *Contd*)

PARTICLE SIZE DISTRIBUTION OF CLAY (FINER PART) FROM DECANTED SUSPENSION

Liquid used : Water 300 ml [Dispersing agent : Sodium hexa meta phosphate (three drops)]
Weight of clay taken = 3 g (Concentration of clay in total suspension = 0·01 mg/cm^3)
Decanted suspension taken in tube = 5 ml
Clay content as determined in the 5 ml of decanted suspension = 0·026 0 g
Room temperature: 27°C
Density of clay = 2·42 g/cm^3
R_1 = 20 cm and R_2 = 21 cm

REV/ MIN	TIME (*Min*)	TUBE No.	WEIGHT OF TUBE (g)	WEIGHT OF TUBE + SAMPLE (g)	WEIGHT OF SAMPLE (g)	PARTICLE SIZE MICRON	MEAN SIZE MICRON	WEIGHT OF PARTICLES (g)	DEPTH FROM FREE SURFACE h_x cm*	CORRECTED WEIGHT OF PARTICLES (g)	PER-CENTAGE†
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)
						3·091‡					
							1·946	0·003 7	7·13	0·006 05	12·3
500	5	1	70·466 5	70·470 2	0·003 7	0·802					
							0·685	0·000 5	0·88	0·000 53	1·1
500	10	2	70·394 9	70·391 9	0·004 2	0·567					
							0·515	0·001 0	0·50	0·001 03	2·1
500	15	3	70·242 7	70·247 9	0·005 2	0·463					
							0·395	0·003 2	0·29	0·003 26	6·5
500	30	4	71·002 2	71·010 5	0·008 3	0·327					
							0·297	0·001 2	0·17	0·001 21	2·4
500	45	5	71·156 6	71·166 2	0·009 6	0·267					
							0·249	0·000 3	0·12	0·000 30	0·6
1 000	15	6	70·861 2	70·871 1	0·009 9	0·231					
							0·197	0·007 6	0·07	0·007 64	15·3
1 000	30	7	70·813 9	70·831 4	0·017 5	0·164					
							0·149	0·003 0	0·04	0·003 00	6·0
1 000	45	8	70·877 6	70·898 1	0·020 5	0·134					
							0·125	0·001 1	0·03	0·001 10	2·2
1 000	60	9	71·223 2	71·264 8	0·021 6	0·116					
							0·058	0·004 4§		0·004 40§	8·8
					0·026 0‖						57·3

NOTE 1 — Cumulative percentage of particles below 3·091 μ is 57·3.

NOTE 2 — The value 7·00 cm in the first row of col 10 is obtained as follows (refer eq. **8.7.2**):
$(h_x/h_o) = (d_x^2/d_o^2)$ or $h_x = (d_x^2/d_o^2)\,h_o = [(1{\cdot}946)^2/(3{\cdot}091)^2]\,18 = 7{\cdot}13$ cm

Corrected weight W_{xe} of particles of mean diameter 1·946 is 0·006 5 g. This is shown in the first row of col 11 and is obtained as follows:

$w_{xe} = w_x h_o/(h_o - h_x) = 0{\cdot}003\,7 \times 18/(18-7{\cdot}13) = 0{\cdot}006\,5$
[Note that w_x = 0·003 7 g is the observed weight (*See* row 1, col 9)].

NOTE 3 — Calculated value of clay in sec of decanted solution is 0·029 g which is 58 percent of 0·05 gm. The observed difference of 0·003 g is due to missing of particles after a lapse of time taken by particle of size 3·091 μ to settle below 18 cm from the free surface from the start of sedimentation.

*The depth from the free surface of a column of suspension from which the mean particle size in the same row in col 8 will be missing after a lapse of time taken by particle of size 3·091 μ to settle below 18 cm from the free surface from the start of sedimentation.

†Percentage taking clay content of undecanted suspension as 100 percent is 0·050 0 g/5 cc (or 3 g of clay in 300 ml).

‡Size of smallest particle found by pipette method.

§Weight of unsettled particles.

‖Total weight of clay [particles in 5 ml of the sample (decanted sample)].

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APPENDIX F

(*Clause* 9.1)

CORRELATION OF RESULTS FROM DIFFERENT METHODS OF SIZE DETERMINATION

F-1. GENERAL

F-1.1 The series of specifications of Indian Standard test methods for powder properties are relevant to particles smaller than 1 000 microns in size. A number of methods (sieving, sedimentation, elutriation, the microscope) are specified for the determination of particle size. No single method is applicable to the whole range of sizes 1 000-1 microns so that, if a powder being analysed is composed of particles of a wide range of sizes, it is necessary to combine the analysis by two or more different methods in order to establish the distribution in full. In general a particle size determination will consist of a sieving of the material down to 75 microns combined with an analysis of the fraction below 75 microns either by sedimentation or elutriation or by microscope.

F-2. FACTORS AFFECTING RESULTS FROM DIFFERENT METHODS

F-2.1 In a particle size determination the particle size is not measured directly but some size-dependent property of the particle is assessed and from this the 'size' of the particle is deduced. This is true even for the microscope method where it is the size of an enlarged image of the particle that is assessed and not the size of the particle itself; the distinction is not important, however, except for sizes near the limit of resolution of the optical system. In sieving, a particle is classified by its ability to pass through an aperture of given size, in sedimentation by its free-falling speed through a stationary fluid, and in elutriation by the upward velocity of a fluid necessary to prevent the particle falling.

F-2.2 If a particle is spherical there is no difficulty in relating the results obtained by different methods of size determination. Because of its symmetry a sphere appears of the same size whatever its orientation and this size is defined completely by its diameter. The diameter is easily measured under the microscope; the limiting diameter of sphere that will pass through a given sieve aperture is well-defined; and Stoke's law relates the free-falling speed of a sphere measured either by sedimentation or by elutriation to its diameter. The same diameter will be assigned to a sphere whatever the method of determination and results obtained by different methods may be combined directly.

F-2.3 Although an irregular particle has a definite volume and surface area the particle cannot be defined in geometric terms and one cannot assign to it a diameter (or a length, breadth and thickness) that enables its volume or surface area to be accurately determined. The size-dependent properties of the particle can still be determined and it is often convenient to represent the particle not by the measured property but by a 'size'. It is customary for the 'size' chosen to be the diameter of the sphere that exhibits the same property as the particle, that is the sphere that has the same projected area, or that just passes through the same sieve aperture or that falls at the same speed through a fluid. However the sphere that is equivalent to a particle in regard to one size-dependent property (say, projected area) is not necessarily of the same diameter as the sphere equivalent in regard to some other property (say, free-falling speed). An irregular particle, therefore, is assigned different 'sizes' by different methods of determination and results obtained by different methods cannot be combined directly. A further point is that the size-dependent properties of a non-spherical particle depend on its orientation so that its 'size' even as determined by a single method is no longer a constant of the particle.

F-2.4 The discrepancy between the 'sizes' assigned to a particle by different methods depends on the departure of the particle from sphericity. It is possible to combine the 'sizes' from different methods to give non-dimensional parameters known as shape-factors which serve to characterize the shape of the particle. A single shape-factor does not characterize a particle exclusively and the particle has as many shape-factors as there are pairs of methods of determining its size. In general, especially in the sub-sieve range, information is obtained not on the sizes of individual particles but on the proportions either by number or by weight, of particles in a series of size classes. Shape-factors are usually, therefore, the ratio for a group of particles of the mean 'sizes' determined by two different methods.

F-2.5 Three situations demanding a knowledge of shape-factors may arise as given below:

a) It may be necessary to convert from a size distribution by number to one by weight, or *vice-versa*. The conversion is usually made by assuming that particles of all sizes have the same shape in the sense that the volume of the particle is proportional to the cube of the measured diameter. It is further assumed that all particles have the same density. If particles of all sizes are not of the same shape, the relationship of measured diameter to volume has to be known for each size class.

b) It may be necessary to estimate some size-dependent property of a powder where it cannot be determined directly.

c) It may be necessary to combine size distribution determined by different methods.

F-3. CORRELATION FACTORS FOR DIFFERENT METHODS

F-3.1 To assist in the situations given in **F-2.5** (b) and **F-2.5** (c), recommended factors are shown for use, in the absence of specific knowledge, in correlating the following particle diameters:

a) Sieve — nominal sieve aperture through which particle just passes.

b) Projected — diameter of circle of area equal to the projected area of particle resting in its most stable position.

c) Stokes — diameter of sphere of equal density having the same free-falling velocity as the particle in a fluid under identical conditions within the range of Stoke's law.

d) The conversion values are given below:

To convert	*Multiply by*
Sieve to projected	1·40
Sieve to Stokes	0·94
Projected to sieve	0·71
Projected to Stokes	0·67
Stokes to sieve	1·07
Stokes to projected	1·50

Example:

The mean diameter of particles which would just pass a 75-micron sieve aperture would match a 106-micron diameter circle by the microscope, and their mean Stokes diameter would be 70·5 microns.

F-3.2 The factors should be applied with caution especially if there is any suspicion that the particles of the powder under test are of extreme shapes, for example, if they have cleavage planes or are acicular. It is highly preferable to use correlation factors specifically determined for the powder. Possible methods of establishing factors are:

a) to overlap the methods of size determination so that one or more size classes are assessed by both methods;

b) to size a sample of the material similar to the sample under test by both methods of size determination.

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